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GLASS FIBER RESIN COMPOSITES AND COMPONENTS AT ARCTIC TEMPERATURES

by

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June 2015

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**GLASS FIBER RESIN COMPOSITES AND COMPONENTS AT ARCTIC
TEMPERATURES**

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ABSTRACT

Glass fiber reinforced composites (GFRC) are used in a wide variety of applications within the U.S. Navy. With a potential increase in arctic operations in the Navy's future, it is important to understand how GFRCs will react at arctic temperatures. Previous research has shown that material properties of GFRC at cold temperatures are dependent on the reaction between the fiber and the resin, but little research has been conducted that was geared toward naval applications at arctic temperatures. This thesis focuses on the tensile properties of GFRC, resin, and glass fiber used in previous NPS-related composite research. The properties of the individual components are compared to assist in the design of composite structures, and provide a baseline to assess the need to re-conduct previous composite experiments at arctic temperatures.

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TABLE OF CONTENTS

I.	BACKGROUND	1
A.	NAVY INTERESTS.....	1
	1. The Arctic	1
	2. Prior Research.....	1
B.	TERMINOLOGY	1
	1. Stress	1
	2. Strain.....	1
	3. Young's Modulus	2
C.	ARCTIC TEMPERATURE EFFECTS ON COMPOSITES	2
D.	OBJECTIVE	2
II.	EXPERIMENT	3
A.	COLD CHAMBER	3
	1. Cold Chamber Construction.....	4
	2. Insulation	4
	3. Temperature Control.....	4
	4. Table.....	4
B.	FIBER SAMPLE.....	5
	1. Fiber Preparation.....	5
	2. Fiber Holder	5
	a. Attempt 1.....	6
	b. Attempt 2.....	6
	c. Successful Holder	7
	d. Recommended Holder.....	7
	3. Fiber Wrapping Procedure.....	8
C.	RESIN SAMPLE.....	8
	1. Specimen Mold	8
	a. Bottom Plate	8
	b. Middle Plate.....	9
	c. Top Plate.....	10
	2. Specimen Mold Preparation	10
	a. Mold Leak Prevention.....	10
	b. Mold Assembly	10
	c. Mold Surface Preparation	11
	3. Resin Specimen Manufacturing.....	12
	a. Resin Mixing	12
	b. Resin Application	12
	c. Curing.....	13
	d. Specimen Extraction	14
	4. Specimen Protection	15
D.	COMPOSITE	15
	1. Fabrication.....	15
	2. Tabs, Adhesives, and Specimen Size	16

a.	<i>Attempt 1</i>	16
b.	<i>Attempt 2</i>	16
c.	<i>Attempt 3</i>	17
d.	<i>Attempt 4</i>	17
E.	TENSILE TESTING	17
III.	RESULTS	19
A.	FIBER	19
1.	Stress Versus Strain of Fiber	19
2.	Ultimate Strength of Fiber	20
3.	Fracture Strain of Fiber	21
4.	Young's Modulus of Fiber.....	22
B.	RESIN	23
1.	Visual Inspection	23
2.	Stress Versus Strain of Resin	24
3.	Ultimate Strength of Resin.....	25
4.	Fracture Strain of Resin	26
5.	Young's Modulus of Resin	27
C.	COMPOSITE	28
1.	Visual Inspection of Composite	28
2.	Stress Versus Strain of Composite	28
3.	Ultimate Strength of Composite	29
4.	Fracture Strain of Composite	30
5.	Young's Modulus of Composite.....	31
D.	TEMPERATURE CONTROL	32
E.	DISCUSSION	33
1.	Data Distortion	33
a.	<i>Temperature Effects</i>	33
b.	<i>Manufacturing Variance</i>	33
2.	Anomalies and Theories	34
a.	<i>Theory 1: Quiet Epoxy Failure</i>	34
b.	<i>Theory 2: MTS Grip</i>	35
c.	<i>Theory 3: Accidental Damage</i>	35
d.	<i>Theory 4: The Data is Correct</i>	35
IV.	CONCLUSION	37
A.	REPEATABILITY	37
B.	RECOMMENDATIONS.....	37
C.	FUTURE RESEARCH.....	37
	APPENDIX A. RESIN SPECIMEN FABRICATION	39
	APPENDIX B. COMPOSITE SPECIMEN.....	41
	LIST OF REFERENCES	43
	INITIAL DISTRIBUTION LIST	45

LIST OF FIGURES

Figure 1.	Cold chamber.	3
Figure 2.	First fiber holder.	6
Figure 3.	Fiber setup with successful holder.	7
Figure 4.	Underside view of bottom plate.	9
Figure 5.	Middle plate.	9
Figure 6.	Top plate.	10
Figure 7.	Assembled mold.	11
Figure 8.	Resin-filled mold.	13
Figure 9.	Mold curing setup.	13
Figure 10.	Specimen release setup.	14
Figure 11.	Resin protection.	15
Figure 12.	Complete resin specimen.	15
Figure 13.	First composite specimen attempt.	16
Figure 14.	Second composite specimen attempt.	16
Figure 15.	Third composite specimen attempt.	17
Figure 16.	Forth composite specimen attempt.	17
Figure 17.	MTS cold setup.	18
Figure 18.	Stress versus strain of fiber at room temperature.	20
Figure 19.	Stress versus strain of fiber at cold temperature.	20
Figure 20.	Typical failure of a resin specimen at room temperature.	23
Figure 21.	Typical failure of a resin specimen at cold temperatures.	23
Figure 22.	Stress versus strain of resin at room temperatures.	25
Figure 23.	Stress versus strain of resin at cold temperatures.	25
Figure 24.	Stress versus strain of composite at room temperatures.	29
Figure 25.	Stress versus strain of composites at cold temperatures.	29
Figure 26.	Sample temperature change over course of fiber test.	33
Figure 27.	Comparison of room and cold composite stress versus strain graphs.	35

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LIST OF TABLES

Table 1.	Comparison of ultimate tensile strength of fiber.	21
Table 2.	Comparison of fracture strain of fiber.	22
Table 3.	Comparison of Young's modulus of fiber.	22
Table 4.	Comparison of ultimate tensile strength of resin comparison.	26
Table 5.	Comparison of fracture strain of resin.	27
Table 6.	Comparison of Young's modulus of resin.	27
Table 7.	Comparison of ultimate tensile strength of composite.	30
Table 8.	Comparison of fracture strain of composite.....	31
Table 9.	Comparison of Young's modulus of composite.	32

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LIST OF ACRONYMS AND ABBREVIATIONS

Δl	Change in length
A	Cross sectional area
F	Force
GFRC	Glass fiber reinforced composites
NPS	Naval Postgraduate School
l_0	Original length
ϵ	Strain
Σ	Stress
MTS	Tensile test equipment used in experiment
USN	United States Navy
E	Young's modulus

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I. BACKGROUND

A. NAVY INTERESTS

1. The Arctic

With the Arctic Ocean's ice retreating, the United States Navy (USN) has begun making preparations for conducting Arctic operations. Part of these preparations involves the study of requirements and capabilities necessary to operate in the Arctic [1]. The Arctic routinely sees temperatures as low as -30° C during the winter months.

2. Prior Research

The Naval Postgraduate School (NPS) has conducted extensive research of a composite patching used to fix cracking in the superstructures of Ticonderoga class cruisers caused by aluminum sensitization [2], [3], [4]. This research has included a variety of tests to measure the performance of the patch and effectiveness of placing sensors into the patch to monitor crack propagation. All of the testing, however, has been conducted exclusively at room temperatures, and as a result the performance of the patch at arctic temperatures is unknown.

B. TERMINOLOGY

1. Stress

Stress is the force per area and can be calculated using equation 1.

$$\sigma = \frac{F}{A} \quad (1)$$

2. Strain

Strain is the change in length per original length and can be calculated by Equation 2.

$$\epsilon = \frac{\Delta l}{l_0} \quad (2)$$

3. Young's Modulus

Yong's modulus is the constant of proportionality in Hook's Law as shown in Equation 3.

$$\sigma = E\epsilon \quad (3)$$

C. ARCTIC TEMPERATURE EFFECTS ON COMPOSITES

Based off previous research GFRCs act like most materials by showing an increase in the Young's modulus and decrease in ultimate strength at arctic temperatures. This research has also shown that composites show signs of degradation of Young's modulus if cyclic loads are applied at low temperatures at strain rates above 0.001 [5]. This degradation is the result of fiber matrix hardening, micro cracks, and degradation that is caused by thermal stresses. These thermal stresses are caused by the resins and fibers having different thermal expansion coefficients. These stresses are mitigated by more flexible resins that are more conducive to plastic deformation [6].

Most of the previous research involving GFRCs at NPS has involved the use of PRO-SET M1002 resin and 237 hardener. This resin requires baking to fully cure, but most of experiments did not conduct this step. PRO-SET states that this will cause the resin cure to a brittle b-phase, which would be affected by arctic temperatures [7]. No data on the performance of the b-phase cure at arctic temperatures could be found.

D. OBJECTIVE

The objective of this thesis:

1. The study of material behavior of constituent materials of composites such as fibers and resin at temperature in arctic environment
2. The study of how the constituent materials influence the composite material's performance.

II. EXPERIMENT

To establish whether it would be beneficial to revisit prior research at arctic temperatures, tensile testing was conducted on E-glass fiber, resin, and GFRC both at room and arctic temperatures.

A. COLD CHAMBER

A cold chamber was needed to simulate arctic temperatures. Dr. Chanman Park designed a stainless steel chamber (see Figure 1) that would be filled with liquid nitrogen to simulate arctic temperatures.

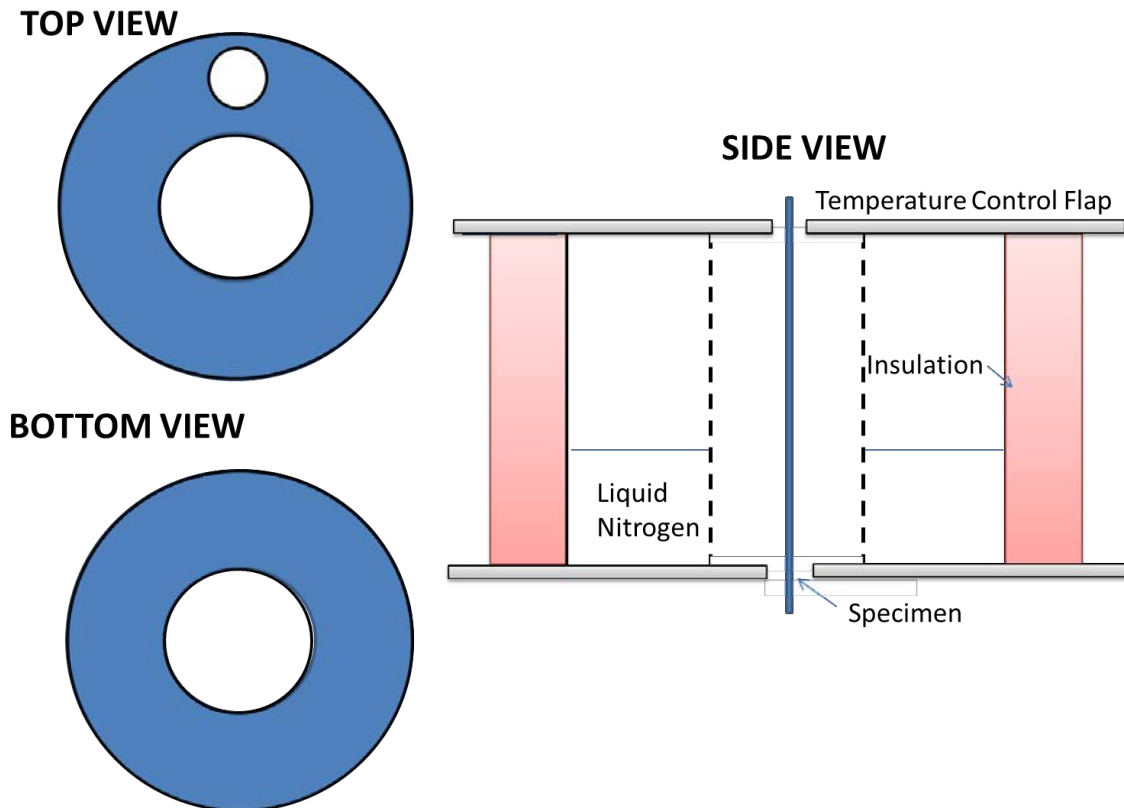


Figure 1. Cold chamber.

1. Cold Chamber Construction

The chamber was a cylinder constructed of stainless steel 3.175 mm thick. It consisted of two cylinders was connected by plates on top and bottom to form a cavity for liquid nitrogen. It had an inner diameter of 50.8 mm outer diameter of 152.4 mm, and was 101.6 mm high. The top plate had a 25.4 mm diameter hole for inserting liquid nitrogen.

2. Insulation

The chamber was originally covered with 88.9 mm Fiberglas wall insulation on the sides and top, but this set-up proved too tall for use. To lower the height, the top insulation was removed and replaced with balsa wood. This resulted in higher temperatures in the center of the chamber, so a foil Mylar rescue blanket was wrapped around the entire chamber to further insulate the chamber.

3. Temperature Control

Maintaining a constant temperature in the center of the chamber proved problematic in initial tests. To control the temperature, pieces of cardboard were wrapped in foil Mylar rescue blanket, and were used to control the size of the chambers hole on the bottom and top. Changing the size of the bottom hole would result in lower temperatures as the size was decreased. Decreasing the size of the top hole would decrease the temperature gradient from top to bottom. Typically, when the chamber cavity was completely full of liquid nitrogen, the bottom whole would be partially open and the top hole would be closed. Closing the top hole would result in a smaller temperature gradient within the chamber as well, but there was still about a 20 °C gradient from top to bottom when the chamber was full. Gradients of 40+ °C were observed with no hole coverage.

4. Table

The tensile test equipment (MTS) used in experiment was not designed to hold the cold chamber. To allow for the chamber to fit with the MTS, a small table was constructed. This table was constructed of plywood and wood 2x4s. The table top

consisted of 254 mm by 305 mm. by 13 mm plywood with a 64 mm hole cut into the center. Four 254 mm pieces of 2x4 were then attached to the bottom of the plywood for use as legs. Additional pieces of 2x4 were added to the sides of the legs to increase stability of the table.

B. FIBER SAMPLE

7500 Hexcel 6 ounce plain weave E-Glass fabric was used in both the fiber and the composite tests, because of its availability and previous use at NPS. It has a manufacturer-listed thickness of 0.150 mm and a measured width per fiber strand of approximately 0.826 mm [8]. Each fiber strand consists of multiple fiber strings. Other E-Glass fabrics with thicker weaves have been used in experiments at NPS, but this was not used because of issues that will be discussed later.

1. Fiber Preparation

The E-Glass fabric comes on a 1067 mm wide roll in a weave pattern. To conduct a tensile test of the fabric, individual strands of the fiber had to be separated from the weave. To accomplish this, a 305 mm by 1067 mm piece of fabric piece of fiber was cut from the roll of fiber. Individual 1067 mm fibers strands were gently pulled from the weave. Two strands were taped together to form one. Two strands were used because one strand had a high rate of accidental breakage, two strands broke at approximately double the force as one, and three strands consistently broke at less the three times that of one. The fibers were taped at one end to help ensure an even load distribution.

2. Fiber Holder

If the fiber was placed directly into the MTS's grip it would be sheared by the force exerted by the grip. To prevent this from happening, a special holder was required. The manufacturer of the MTS makes a special holder just for fiber, but obtaining it was cost prohibitive. As a result, one would have to be manufactured on site.

a. Attempt 1

The first attempt at a holder consisted of a 50.8 mm by 50.8 mm by 6.35 mm aluminum block (see Figure 2). A 3.18 mm hole was drilled through the center of one of the 6.35 mm sides and a groove cut into one of the sides parallel to the hole. In addition, all of the corners were rounded off. The fiber would go through the hole, wrap around the block in the side groove, through the hole again, and be pinched between the block and grip. In theory, this design would distribute the load over a long contact patch and prevent shearing of the fiber. In reality, either the machining marks in the hole or the radius of around the edge of the whole sheared the fiber at less than one newton of force.



Figure 2. First fiber holder.

b. Attempt 2

Due to machine shop lead time, any new holder would have to be assembled by hand from parts easily attainable. A standard steel gutter nail was purchased from a local hardware store and was then duct taped to the top of the failed holder. The fiber was wrapped several times around the nail and then placed between the grip and the block. The fiber broke at around 5 N, but consistently broke where the fiber wrapped around the nail. Due to this breakage point it was correctly feared that the nail was causing a stress concentrator in the fiber, and this was causing the fiber to fail prematurely. It was decided that a wider radius around which to wrap the fiber was needed.

c. Successful Holder

Any holder produced had to fit within the 6.35 mm wide grip of the MTS, so this eliminated wrapping the fiber around a pipe. A coupling nut that had an inner diameter slightly larger than the gutter nail and a 25.4 mm to 19.1 mm copper pipe reducer were purchased. Two layers of duct tape were wrapped around the gutter nail, and the coupling nut was threaded onto the duct tape. The reducer, which had a small side inner diameter slightly smaller than the widest part of coupling nut, was then hammered on the coupling nut. To put all the pieces together, Gorilla glue was applied to the small end of the reducer to attach it directly to the nail (see Figure 3).



Figure 3. Fiber setup with successful holder.

d. Recommended Holder

While the final holder design worked on 7500 Hexcel 6 ounce plain weave E-Glass fabric, it broke in attempts to test other fabric used in previous experiments. The torque caused by the fiber rotating around the pipe reducer was too great for the duct tape and Gorilla glue. Both would fail, allowing the pipe reducer to freely move and the fiber to unwind off the reducer. Additionally, the nail had started to flex under the load. Had time permitted, a holder with a similar design with a corresponding clamp would have been machined from two blocks of steel. This would have prevented the end from spinning and reduced the likelihood of fiber slip.

3. Fiber Wrapping Procedure

To ensure consistent results, each fiber was wrapped around the holders using the same procedure. The fiber was wrapped around the small cylinder once and then big cylinder three times ensuring each wrap was on top of the others. The small cylinder wrap was completely covered in duct tape. The big cylinder had a small piece of duct tape placed on side to keep the fiber from unwrapping. The holder was then placed 254 mm away from the first holder and was wrapped in the reverse order of first holder with one exception. The second holder had an extra wrap around the big cylinder. This wrap was the first wrap around the cylinder and was not overlapped by the other three wraps. This was to prevent a stress concentrator caused by the other three wraps pinching the fiber as soon as it made contact with the holder.

C. RESIN SAMPLE

Resin samples were sized in accordance of ASTM D-638, because the resin's manufacturer PRO-SET uses ASTM D-638 for its tensile test. PRO-SET did not list specimen size or shape, so a 203 mm long 3.16 mm thick type I dog bone specimen was used. Step by step direction can be found in Appendix A.

1. Specimen Mold

To create the specimen, a three-piece mold was constructed using steel plates held together with 10–24 machine screws. The bottom plate was 6.35 mm thick with through holes that were counter sunk in the back to allow the plate to lie flat. The middle plate was a 3.16 mm thick and had three holes in the shape of the specimen cut out. It also had 10–24 threaded screw holes to allow for the bottom plate to be attached to the middle plate.

a. Bottom Plate

The bottom plate was a 6.35 mm in thickness. It had 27 through holes for attaching it to the middle plate, and four 10–24 threaded holes in the corners of the plate to be used for guide screws (see Figure 4). All holes were countersunk in the back to allow the plate to lie flat.



Figure 4. Underside view of bottom plate.

b. Middle Plate

The middle plate was 3.18 mm (see Figure 5) thick, because of the size of the MTS grip. The plate had three holes cut out matching the desired shape of specimens. The specimens were 38.1 mm apart and were 38.1 mm from the edge of the plate. The plate had 10–24 threaded screw holes that were all 19.1 mm from the specimen cutouts with the exception of holes along the gage, which remained in line with the rest of the holes. The holes started along the top edge of each specimen and were spaced at 50.8 mm along the side. Additional holes were also drilled 19.05 mm from the top and bottom of each specimen. Four additional 10–24 clearance holes were drilled at each corner of the plate for guide screws to pass through.

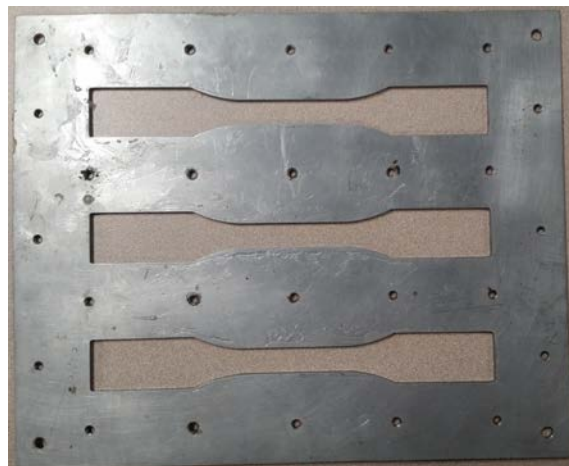


Figure 5. Middle plate.

c. Top Plate

The top plate consisted of 10–24 clearance holes that matched the pattern of the screw pattern of the middle plate (see Figure 6). The top plate was originally intended to ensure a smooth top specimen surface, but early tests showed that the surface was naturally smooth without the top plate. It was needed to prevent the screws from puncturing the plastic vacuum sheet.



Figure 6. Top plate.

2. Specimen Mold Preparation

a. Mold Leak Prevention

Due to warping, the plates do not sit perfectly flush, which would allow the resin to leak in between the plates. Originally, tape was placed over the bottom of the specimen hole and a specimen wax paper cut out was placed over the tape exposed to the resin. This was effective at preventing leakage, but the tape would cause an unacceptable arc to form along the bottom side of the specimen. A gasket made of cardboard with the wax paper glued to the top and screw holes punched out was then made. The gasket successfully prevented leakage and created minimal surface deviation.

b. Mold Assembly

The mold assembly was fastened using 10–24 machine screws (see Figure 7). The four corner guide screws were inserted into the bottom plate first. The cardboard and wax

paper gasket was then placed on top of the bottom plate using the four corner screws as a guide. The middle plate was then placed onto of the gasket in the same manner. Wingnuts were then gently threaded on to the corner screws to keep the two plated together as the remaining screws were installed. The screws were tightened enough to create a seal, but not enough to crush the cardboard. If the screws were over tightened, the cardboard would protrude into the specimen holes causing an uneven specimen surface. Nuts were then threaded on to the four corner screws about 6.35 mm from the top. The nuts were used to support the top plate during curing.



Figure 7. Assembled mold.

c. Mold Surface Preparation

To release the resin specimens from the steel mold after the curing, advance surface preparation was required. Machining of the specimen holes results in small grooves along the sides that would cause the specimen to stick. These grooves were removed with sandpaper. The resin was capable of laminating to the steel mold, so a release agent was required. Initially Meguiar's Mirror Glaze Maximum Mold Release Wax was used, but it did not provide consistent results with the small surface area of the middle plate. It also required multiple layers of wax and each layer took 15–30 minutes to apply correctly. Petroleum jelly was recommended by a faculty member of the NPS Mechanical Engineering Department who was also conducting research with resin

specimen. The petroleum jelly only required one thin coating prior to every use, took less than five minutes to apply, and worked perfectly.

3. Resin Specimen Manufacturing

A mixture of PRO-SET M1002 resin and 237 hardener were used to make the resin specimen. They were used because they were used in prior research conducted at NPS.

a. Resin Mixing

The resin and hardener were measured to the weight ratio of 100:24 for the resin to hardener. For ease of use, three to four times the needed amount of the mixture was made each time. This was done to help reduce the amount of air bubbles in the specimens after the resin mixture was inserted into the mold. The two components were then mixed thoroughly in a plastic beaker. After mixing the mixture, the beaker was then inserted in an ultrasonic bath for five minutes to remove air bubbles from the mixture. During the ultrasonic bath it was normal for a layer of surface bubbles to form. While it was safe to mix the mixture in an adequately ventilated space, the ultrasonic bath caused a rapid discharge of vapors from the mixture, and for safety reasons it was done under a fume hood. Had a fume hood not been available, a respirator should be worn instead.

b. Resin Application

To insert the resin into the mold, a 500 ml syringe was used to minimize resin spilling outside of specimen holes. When inserting the resin into the mold, great caution was used to prevent bubbles from forming in the specimen, but some bubbles would still form. Dragging the tip of the syringe across the bubbles would usually pop them if the mold was not completely full. Due to this, the narrow section of the specimen was filled first, so there would be no air pockets in the critical part of the specimen (see Figure 8). Dipping the syringe in the resin beaker will cause air bubbles to form, but dipping the syringe into the same spot each time and making extra resin minimized the amount of air in the syringe.



Figure 8. Resin-filled mold.

c. Curing

PRO-Set recommends baking the resin at temperatures of 63 °C for 14 hours after initial curing, but since the prior research did not do this, the specimens were cured per prior research under a vacuum of -33.8 kPa at room temperature (see Figure 9).

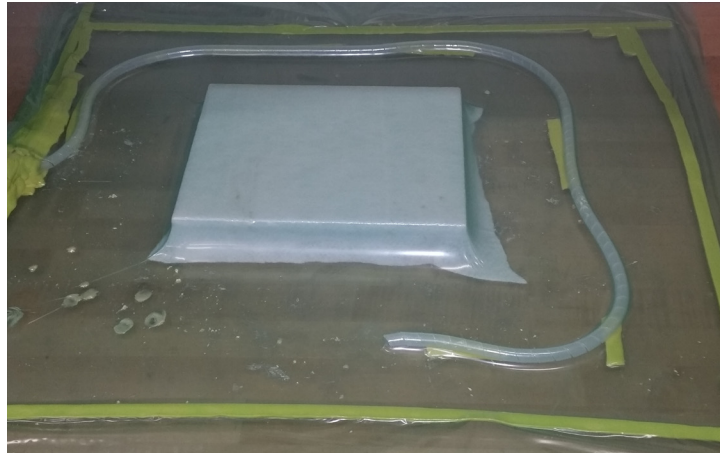


Figure 9. Mold curing setup.

d. Specimen Extraction

Following completion of curing, the mold was turned upside down with the wingnuts still attached to the top of the middle plate. The non-corner screws were then removed and the mold was placed gently in its upright positions. Following removal of the wingnuts, the middle plate and gasket were removed together. The gasket was then gently peeled off the middle plate.

Extracting the specimen from the mold proved difficult in its brittle state. Attempts to push the specimen out by hand resulted in the specimens cracking due to non-uniform pressure. Attempts to push the samples out from the top were slightly more successful, but still resulted in about half of the specimens breaking. Complete extraction success was found using specimens from a previous experiment and heat. First both sides of the mold were heated using a heat gun for about 10 seconds. This reduced friction between the mold and specimen, by expanding the mold and reducing the viscosity of the petroleum jelly. An old dog bone specimen that was left over from a previous experiment was then taped to the back of the new specimen and placed on a table. By pushing on the mold instead of the specimen a more uniform pressure distribution resulted and specimens were extracted (see Figure 10).

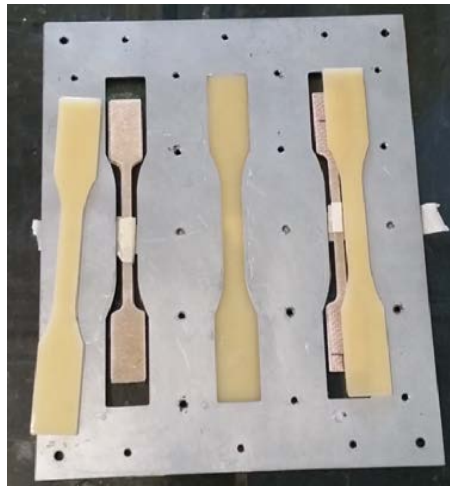


Figure 10. Specimen release setup.

4. Specimen Protection

To prevent the resin specimen from being crushed by the MTS, protection is required. To protect the specimen, a 1.59 mm groove was cut into a 38.1 mm wide by 3.18 mm thick strip of steel (see Figure 11). This was then cut into 25.4 mm long pieces. The groove and specimen were then roughed up with sandpaper and washed. Two strips were then glued to the specimen using Gorilla glue with the groove contacting the specimen (see Figure 12).



Figure 11. Resin protection.



Figure 12. Complete resin specimen.

D. COMPOSITE

1. Fabrication

Composite specimens were fabricated using the steps detailed in Appendix B.

2. Tabs, Adhesives, and Specimen Size

a. Attempt 1

A 279 mm by 25.4 mm composite specimen was fabricated for use in the test. To control the location of the break of the composite, aluminum tabs were adhered to the specimen. These tabs were 25.4 mm by 38.1 mm and attached by resin (see Figure 13). When the specimen was tested, however, the tabs failed to stay adhered to the composite. It was thought the resin was not strong enough, so all the tabs were cut off and the composite strips were reduced to 191 mm.



Figure 13. First composite specimen attempt.

b. Attempt 2

After the resin failed, it was decided to conduct the test with using Gorilla glue to adhere the tabs (see Figure 14). The glue lasted longer than the resin, but still failed before the composite sample broke. This time, however, the sample broke within 85% of the max rating of the MTS used to conduct the test, so it was decided to test this sample again in a tensile tester with a higher rating. The composite sample ended up failing at 140% of the original machine's max rating. In order to keep everything standard with the other test conducted, it was decided to cut the samples down to 12.7 mm.



Figure 14. Second composite specimen attempt.

c. Attempt 3

It was decided to attempt to use the same original tabs with the 12.7 mm samples, and attempting to stabilize them with extra glue, or scrap piece of composite (see Figure 15). This too failed, so it was decided to extend the length of the tabs to 63.5 mm and 12.7 mm wide, and switch to an epoxy.

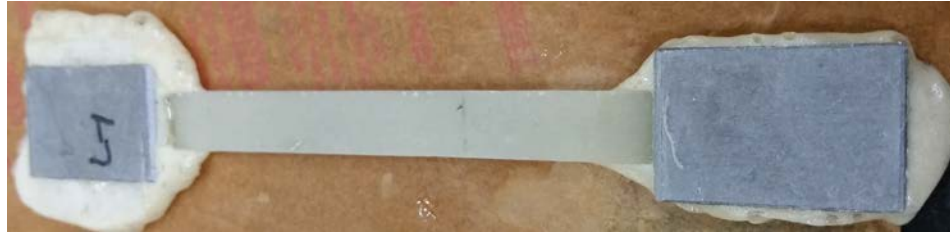


Figure 15. Third composite specimen attempt.

d. Attempt 4

The new tabs were attached to the composite using a two-part marine epoxy by Loctite (see Figure 16). This epoxy was designed to work with aluminum and fiberglass. This setup worked with some cracking in the epoxy at room temperature, but the epoxy failed to control the location of the failure in the cold test.



Figure 16. Forth composite specimen attempt.

E. TENSILE TESTING

The tensile testing was conducted on all samples using the MTS 858 Table Top System. It was set to a rate of 3 mm/min, and the only adjustment made between the different types of specimen was height. For the cold test, the table and cold chamber were placed on the flat area along the bottom of the machine (see Figure 17). The chamber was then partially filled with liquid nitrogen to cool it down to between -25°C and -30°C.

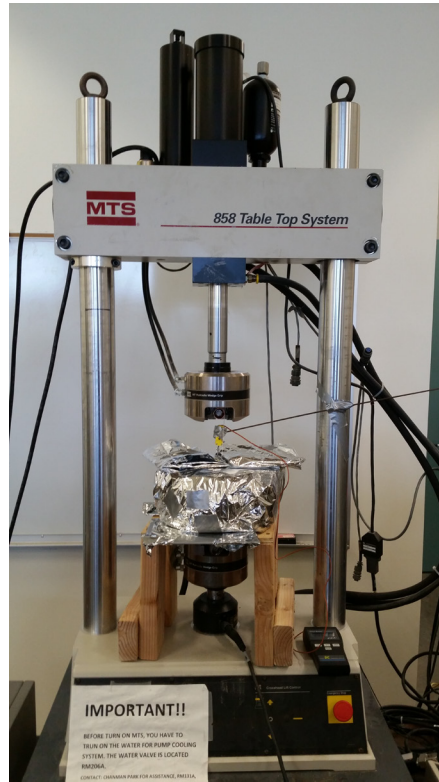


Figure 17. MTS cold setup.

III. RESULTS

A. FIBER

1. Stress Versus Strain of Fiber

The cold and room temperature graphs have data sets with a wide distribution (see Figures 19 and 18). The strain difference can be attributed to the fact the fiber holder uses friction to hold the fiber in place. As a result, the fiber rotates around the holder until the tension in the fiber is high enough to keep the fiber in place. This causes a distortion at the start of the test. For some of the test it is easy to remove this section, but if the fiber is slow to catch it will distort the result. The cold fibers typically experienced more difficulty catching than the room temperature fiber. Another reason is the fact that each test used two strands of fiber. While great care was taken to ensure both fibers carried a uniform load; it is not possible to make the load perfectly balanced. For the room temperature test, the fiber's ability to stretch compensated for this. In the cold test, the increased stiffness of the fiber amplified any difference between the two fibers. The increased stiffness also explains the difficulties getting the cold fibers to catch on the holder. It took a total of 12 cold tests to get the five tests displayed, and two of those tests were less than ideal.

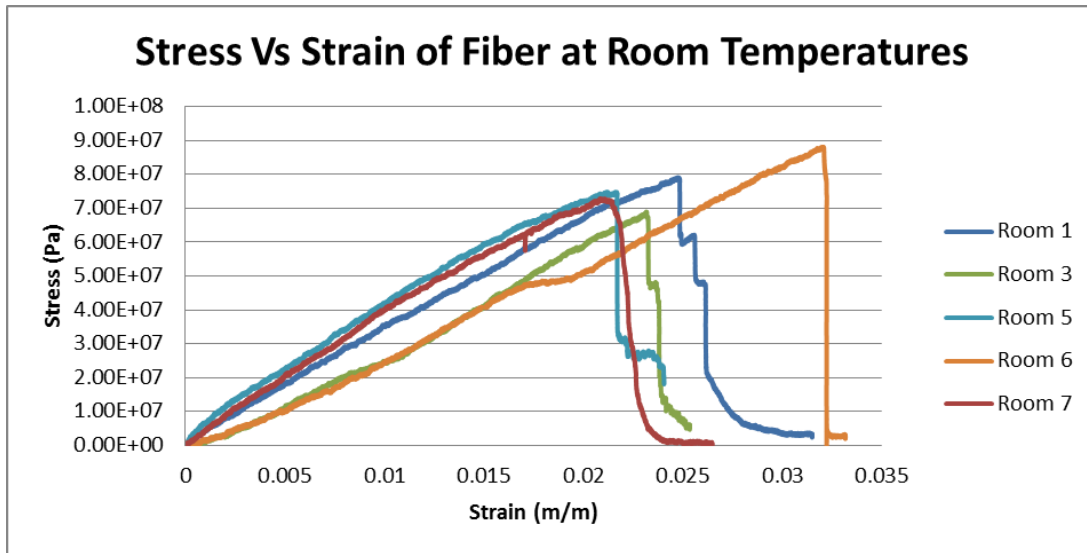


Figure 18. Stress versus strain of fiber at room temperature.

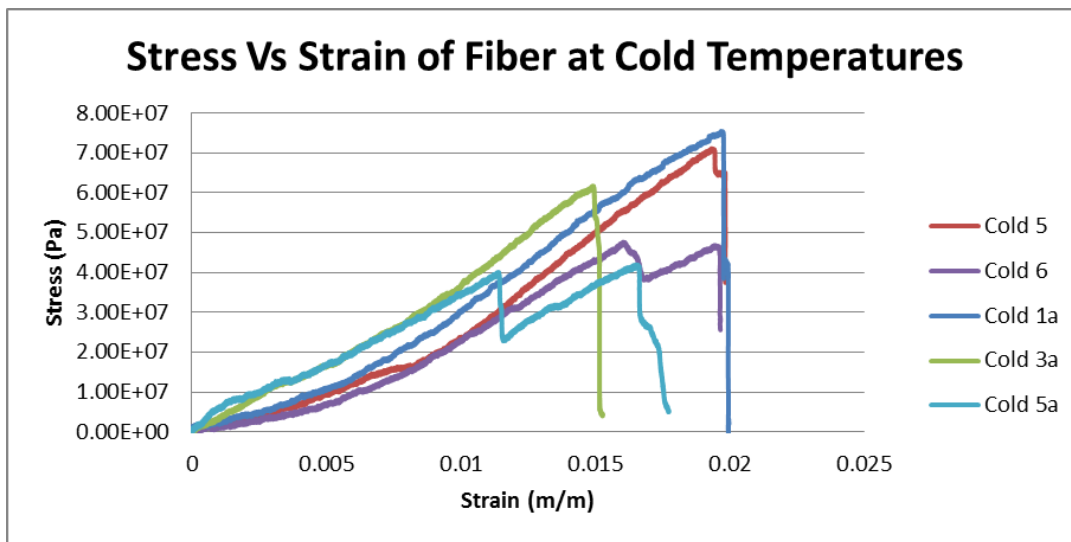


Figure 19. Stress versus strain of fiber at cold temperature.

2. Ultimate Strength of Fiber

Both data sets contained a wide range of ultimate strengths (see Table 1). Some of this could be accounted for by the fact the same cross-sectional area was used for all calculations. This was done due to the difficulty measuring individual strands. Since all of the fibers came from the same e-glass sheet, the areas should have been relatively consistent, but since the cross-sectional area of the fiber was extremely small, any minor

change would be amplified in the end. The cold tests were further amplified by increased sensitivity to mismatches in lengths of fiber strands. As a result, one of the fibers was carrying a greater portion of the load and failing earlier than if the load was uniformly distributed. Removing the non-uniform load early failure, cold testing raised the cold average to 6.49E+07 Pa. Run 6 of the room temperature test appears to be an outlier from the other room temperature test. The fibers in run 6 appear to have slipped slightly in the middle of the test. It is possible that this slip somehow affected the load distribution of the two fibers, causing a near perfect distribution. With run 6 removed, the average decreases to 7.40E+07 Pa. By removing the outlier samples, the difference between room and cold is 0.91E+07 Pa or 12.3%, which would still be outside the standard deviation for room temperature tests.

Table 1. Comparison of ultimate tensile strength of fiber.

Ultimate Strength of Fiber (Pa)			
Room Temperatures		Cold Temperatures	
1	7.91E+07	5	7.12E+07
3	6.90E+07	6	4.76E+07
5	7.48E+07	1a	7.55E+07
6	8.81E+07	3a	6.17E+07
7	7.29E+07	5a	4.20E+07
Average	7.68E+07	Average	5.96E+07
Standard Deviation	6.54E+06	Standard Deviation	1.30E+07
Difference (Pa)	-1.7E+06	Difference (%)	-22.3

3. Fracture Strain of Fiber

With the exception of room temperature run 6, all the room tests had a close data set for fracture strain (see Table 2). When you remove run 6, the average strain drops to 0.023. The cold data had two separate groups form. When you compare those groups to the stress strain graphs, it becomes apparent the higher strain rate samples were also the tests that experienced fiber slippage at the beginning of the test. This slippage would have increased the strain rates of these tests. Without the slippage, the average would probably fall to 0.016 or possibly lower. With the adjusted numbers, the max strain rate difference is around 0.007, but percent difference increased to 30.4%.

Table 2. Comparison of fracture strain of fiber.

Fracture Strain of Fiber (m/m)			
Room Temperatures		Cold Temperatures	
1	0.025	5	0.019
3	0.023	6	0.016
5	0.021	1a	0.020
6	0.032	3a	0.015
7	0.021	5a	0.017
Average	0.024	Average	0.017
Standard Deviation	0.004	Standard Deviation	0.002
Difference (m/m)	-0.007	Difference (%)	-29.2

4. Young's Modulus of Fiber

Young's modulus of the fiber test is an excellent way to tell the difference between the room and cold test (see Table 3). It can be calculated anywhere along the stress strain graph, so calculations can be taken where it is known that the fiber is not slipping. As a result, the calculated values are very close together, and the data outliers in room temperature disappear. Young's modulus also confirms the fact the runs 6 and 5a were not carried uniformly. This reduced Young's modulus of both, because one fiber is not as stiff as two. They were left in the average, because the fibers are actually used in a cloth. If the fibers are unable to flex enough to distribute the load, the cloth would reduce rigidity. Even with the two lower data points included, the average Young's modulus of the cold fiber test is 53.2% greater than the room temperature test.

Table 3. Comparison of Young's modulus of fiber.

Young's Modulus of Fiber (Pa)			
Room Temperatures		Cold Temperatures	
1	2.93E+09	5	4.50E+09
3	2.86E+09	6	3.96E+09
5	2.77E+09	1a	4.51E+09
6	2.83E+09	3a	4.60E+09
7	2.80E+09	5a	4.19E+09
Average	2.84E+09	Average	4.35E+09
Standard Deviation	5.37E+07	Standard Deviation	2.40E+08
Difference (Pa)	1.51E+09	Difference (%)	53.2%

B. RESIN

1. Visual Inspection

The resin samples exhibited signs of brittleness in how they broke. The breaks were not clean and consistently resulted in pieces missing from the center following failure. In one case, a piece was exhibited to fly approximately 3 m following failure. It appears that the missing piece was bigger on the cold samples (see Figure 20) than on the room temperature samples (see Figure 21).



Figure 20. Typical failure of a resin specimen at room temperature.



Figure 21. Typical failure of a resin specimen at cold temperatures.

2. Stress Versus Strain of Resin

The stress versus strain graph of resin at room temperature shows two distinct failure groups forming on the graph (see Figure 22). Specimens 3-1 and 3-2 failed at strains that are about .005 m/m less than the remaining three samples, and also have lower ultimate strength. Specimen 3-3's ultimate strength was closer to 3-1's and 3-2's, but it fractured at a strain rate closer to 1-1 and 4-1. The low ultimate tensile strength of all of the batch 3 samples indicates that something in the resin mix is affecting ultimate tensile strength. Closer examination of specimen 3-2 revealed a spot of glue next to the fracture. This could have potentially caused a stress concentrator and resulted in the sample failing early. Examination of 3-1 revealed a small chip near the fracture, but not at the fracture point. While small chips were formed along the edges during extraction from the mold of most samples, this one was slightly larger than the rest. It is possible that there was an even larger chip causing 3-1 to fail early.

The stress versus strain diagram for the cold temperatures test shows a strong grouping of test, with one outlier (see Figure 23). Three of the four tests actually sit virtually on top of one another. Sample 5-1 has a slightly higher ultimate strength, but had a fracture strain near the three specimens that were virtually on top of each other. Specimen 5-3 result is significantly higher than the rest of the cold specimens tested. Sample 5-3 actually has the highest yield strength of all specimen tested, but the only the fourth highest fracture strain. Upon further inspection, specimen 5-3 showed no signs of chips from being removed from the mold, while every other specimen tested showed some small chips. In addition, specimen 5-3 was the last sample tested, with the least amount of liquid nitrogen remaining in the cold chamber. While the chamber was cooled to the required temperature, it required the temperature control flaps to be closed almost fully. The other tests were conducted with the flaps about one-third open to keep the chamber from being outside of the desired temperature range. This could have possibly affected the temperature distribution of the specimen, causing specimen 5-3 to be slightly warmer than the rest.

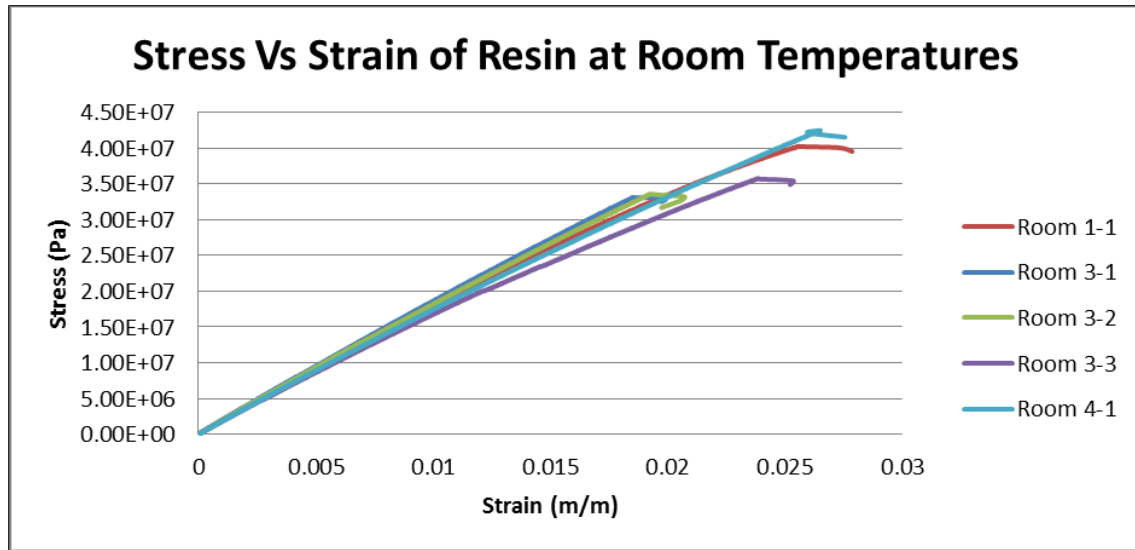


Figure 22. Stress versus strain of resin at room temperatures.

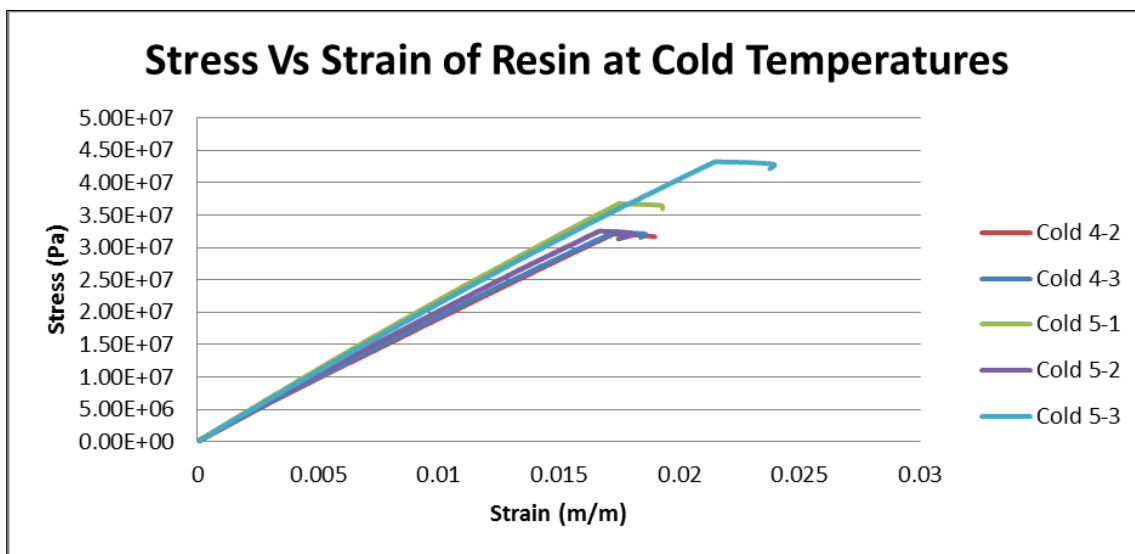


Figure 23. Stress versus strain of resin at cold temperatures.

3. Ultimate Strength of Resin

Based on the ultimate tensile strengths of both the room and cold temperature tests, it would appear the simulated arctic temperatures in the cold test had no significant effect on the ultimate tensile strength of the material (see Table 4). A deeper look into the data, however, shows some tests have potentially distorted the results. It was only possible to make three specimens at the same time, because of pot life of the resin and

size of the mold. The room temperature test was actually made in three separate batches, because two of three specimens in batch 1 broke during extraction. As a result, it becomes clear that something could have potentially gone wrong with batch 3 causing it to be weaker than the others. Sample 3–1 is $9.4\text{E}+6$ Pa lower than the sample 4–1, which is almost three times the standard deviation. Removing sample 3–1 alone raises average strength to $3.80\text{E}+07$ Pa and removing sample 3–2, which is only slightly higher, raises the average strength to $3.95\text{E}+07$ Pa. In the cold data set, it becomes clear that sample 5–3 does not belong. It is $6.45\text{E}+06$ Pa stronger than the nearest cold test, which is also from the same batch. Removing sample 5–3 drops the colds average ultimate strength to $3.35\text{E}+07$ Pa. Removing the distorting tests increases the difference in ultimate tensile strength to $6.04\text{E}+06$ Pa or 15.3%, which is now outside the standard deviation of either test with the removed tests.

Table 4. Comparison of ultimate tensile strength of resin comparison.

Ultimate Strength of Resin (Pa)			
Room Temperatures		Cold Temperatures	
1–1	$4.03\text{E}+07$	4–2	$3.21\text{E}+07$
3–1	$3.31\text{E}+07$	4–3	$3.24\text{E}+07$
3–2	$3.36\text{E}+07$	5–1	$3.68\text{E}+07$
3–3	$3.58\text{E}+07$	5–2	$3.25\text{E}+07$
4–1	$4.25\text{E}+07$	5–3	$4.28\text{E}+07$
Average	$3.70\text{E}+07$	Average	$3.54\text{E}+07$
Standard Deviation	$3.72\text{E}+06$	Standard Deviation	$4.28\text{E}+06$
Difference (Pa)	$-1.63\text{E}+06$	Difference (%)	-4.40

4. Fracture Strain of Resin

Based on the initial data without removing the same outliers as before, the difference between both data sets is already outside the standard deviation (see Table 5). Sample 3–3 was not an outlier as in the strength results, but the remainders of the strength outliers are still outliers. When the outliers are removed, the difference increases even further to 0.0094 m/m or 35.3%. Overall the temperature has a significant effect on fracture strain.

Table 5. Comparison of fracture strain of resin.

Fracture Strain of Resin (m/m)			
Room Temperatures		Cold Temperatures	
1-1	0.0256	4-2	0.0174
3-1	0.0197	4-3	0.0174
3-2	0.0207	5-1	0.0175
3-3	0.0252	5-2	0.0167
4-1	0.0292	5-3	0.0224
Average	0.0241	Average	0.0183
Standard Deviation	0.0035	Standard Deviation	0.0021
Difference (m/m)	-0.0058	Difference (%)	-24.1%

5. Young's Modulus of Resin

The difference in Young's modulus was greater than the standard deviation of either data set with the outliers included (see Table 6). Sample 5-3 fell inside the standard deviation, and did not have the highest Young's modulus. The same could not be said with the sample 3-1 in the room temperature data set, as it fell outside the standard deviation. Sample 3-2 was equal to the deviation, but the standard deviation for the room temperature data set with samples 3-1 and 3-2 removed dropped to 3.39E+07 Pa and the average dropped to 1.56E+09 Pa. It also increased the difference to -3.94E+08 Pa or -25.3%.

Table 6. Comparison of Young's modulus of resin.

Young's Modulus of Resin (Pa)			
Room Temperatures		Cold Temperatures	
1-1	1.57E+09	4-2	1.85E+09
3-1	1.79E+09	4-3	1.86E+09
3-2	1.74E+09	5-1	2.10E+09
3-3	1.50E+09	5-2	1.95E+09
4-1	1.60E+09	5-3	2.01E+09
Average	1.64E+09	Average	1.95E+09
Standard Deviation	1.06E+08	Standard Deviation	9.30E+07
Difference (m/m)	3.1E+08	Difference (%)	18.9

C. COMPOSITE

1. Visual Inspection of Composite

Both the cold and room temperature samples were affected by the difference in stiffness between the aluminum tabs and GFRC specimens. This caused stress along the contact patch, and as a result epoxy that holds the two the surfaces together cracked. The room temperature specimens were affected less, as all samples still fractured either within the specimen's gage area or within 1 cm of the gage area. The epoxy failed completely with the cold specimen, and all specimens broke where the MTS clamped onto the specimen.

The breaks within the room temperature specimens appear to be relatively clean, straight, and showed some signs of delamination and deformation near the fracture. The fracture within the cold specimen had jagged edges with fibers hanging out. Due to the fracture occurring inside of the holder region epoxy obstructs the view of deformations and delamination, so not direct comparison between room and cold breaks can be made.

2. Stress Versus Strain of Composite

The epoxy cracking severely distorted the strain rates and diminished the usefulness of stress versus strain graphs (see Figures 24 and 25). A general idea of the stress strain curve can be obtained, however. Both appear to have a larger elastic region than that of resin or fiber. The room temperature tests also appear to have a tight group in terms of strength. Both graphs show signs of either the epoxy cracking or individual fibers starting to fail within the composite.

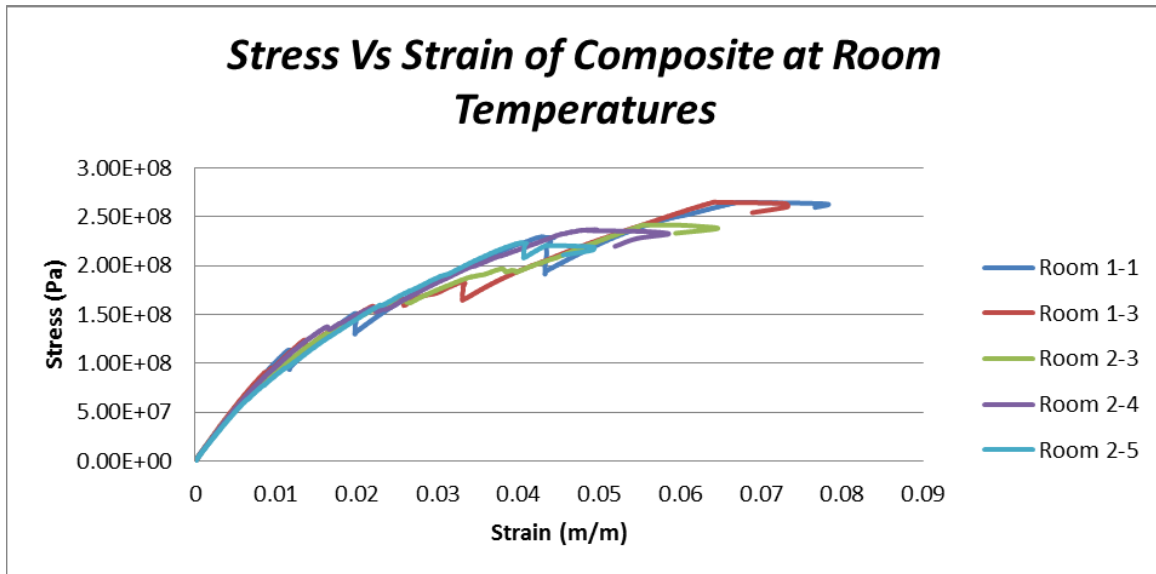


Figure 24. Stress versus strain of composite at room temperatures.

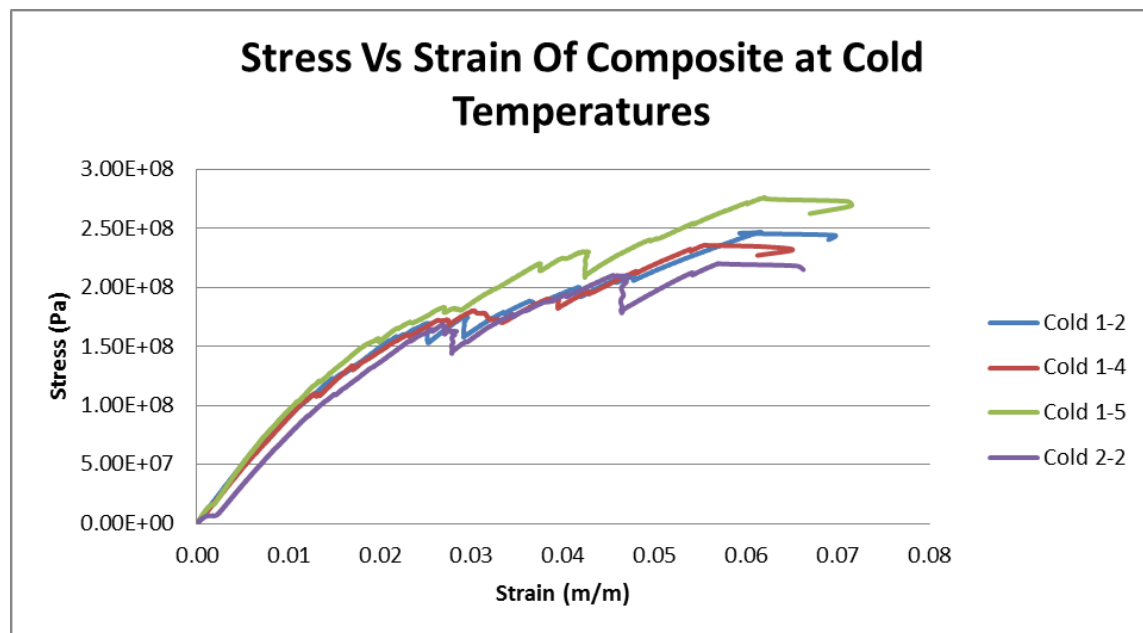


Figure 25. Stress versus strain of composites at cold temperatures.

3. Ultimate Strength of Composite

There is no significant difference between the ultimate strength of the room and cold tests (see Table 7). The only potential outliers are samples 2-5 and 1-5. Due to earlier difficulties with getting the composite tensile test to work at room temperature,

both samples 1–5 and 2–5 were made from scrap pieces. As a result both were slightly skinner than the rest and the edges were sanded post cut by hand instead of machine. This could explain why sample 2–5 failed early, but not why sample 1–5 was stronger than the rest of the cold samples. If sample 2–5 was removed the average increases to $2.52\text{E}+08$ Pa. Removing sample 1–5 would decrease the cold average to $2.34\text{E}+08$ Pa. This increases the difference between the two data sets to $1.8\text{E}+07$ Pa, which is outside the standard deviation of the room temperature test. Batch1 samples appear to have a higher strength than batch 2, which can distort the results since the cold test are batch 1 heavy.

Table 7. Comparison of ultimate tensile strength of composite.

Ultimate Strength of Composite (Pa)			
Room Temperatures		Cold Temperatures	
1–1	2.65E+08	1–2	2.47E+08
1–3	2.65E+08	1–4	2.36E+08
2–3	2.42E+08	1–5	2.76E+08
2–4	2.37E+08	2–2	2.20E+08
2–5	2.23E+08		
Average	2.46E+08	Average	2.45E+08
Standard Deviation	1.63E+07	Standard Deviation	2.05E+07
Difference (Pa)	-0.01E+08	Difference (%)	-0.4%

4. Fracture Strain of Composite

The strain data for both room and cold tests is useless (see Table 8). There is no way to accurately say what the length of the specimen is after the epoxy starts to crack, and because of this there is no basis to compare the two. The strong grouping of cold is significant since the epoxy failed completely, so the lengths for all the cold test samples were very close at the end, but there is still no way to compare them to the room temperature tests.

Table 8. Comparison of fracture strain of composite.

Fracture Strain of Composite (m/m)			
Room Temperatures		Cold Temperatures	
1-1	0.078	1-2	0.069
1-3	0.070	1-4	0.065
2-3	0.060	1-5	0.071
2-4	0.052	2-2	0.066
2-5	0.048		
Average	0.061	Average	0.068
Standard Deviation	0.011	Standard Deviation	0.002
Difference (m/m)	0.007	Difference (%)	11.4

5. Young's Modulus of Composite

Even with the distorted strains experienced in this test, Young's modulus can still be calculated in the area prior to the epoxy cracking (see Table 9). The layer of epoxy applied to the samples between the metal taps and composite strip was thin enough to prevent a significant distortion prior to cracking. The room temperature data set shows a relatively small deviation in data. The batch 1 specimens did have a slightly higher Young's modulus than batch 2. The deviation in cold data was great, but the trend of batch 2 being lower than batch 1 continued. Specimen 2-1 was also included in cold data even though it was not included in the other sections. Specimen 2-1 was completely pulled out of the holders when the initial test was conducted, but still provided data up to the epoxy failure. It was included because the cold data set was batch 1 heavy and the room data set was batch 2 heavy. Adding 2-1 helps mitigate differences between the batches in the final data. The composite Young's modulus data did produce an interesting effect. The Young's modulus decreased by $7.60\text{E}+08$ Pa or 7.45% when the sample were cooled down.

Table 9. Comparison of Young's modulus of composite.

Young's Modulus of Composite (Pa)			
Room Temperatures		Cold Temperatures	
1-1	1.02E+10	1-2	9.73E+09
1-3	1.06E+10	1-4	9.40E+09
2-3	1.00E+10	1-5	9.96E+09
2-4	1.00E+10	2-2	9.08E+09
2-5	9.95E+09	2-1*	9.06E+09
Average	1.02E+10	Average	9.44E+09
Standard Deviation	2.37E+08	Standard Deviation	3.12E+08
Difference (Pa)	7.60E+08	Difference (%)	7.45

D. TEMPERATURE CONTROL

The cold chamber design proved problematic for controlling temperature. When completely full of liquid nitrogen, the temperature difference between top and bottom of the chamber was recorded to be as high as 50 °C. While this was mitigated by only filling the chamber up about one-third full, this still resulted in up to a 15 °C. Adding a cover to the top further reduced the gradient to 10 °C. There was also a gradient from side to center of the chamber. The sides of the cold chamber were under -60 °C, but with no covers on the top or bottom, a temperature of 10 °C would be recorded. This made obtaining accurate measurements of the temperature inside the chamber difficult. The specimen being tested was always directly in the center of the chamber, but two objects cannot occupy the space, and the thermal couple was always off center. This resulted in difficulties obtaining accurate measurements of the temperatures that the specimens were being exposed to. To further complicate matters, the temperature in the chamber was controlled by opening and closing sliding flaps along the bottom of the chamber. If the flaps were partially closed beneath the thermal couple, but not the next to the specimen, the temperature difference would increase further.

Temperature stability was also an issue during the experiments. The temperatures during the course of the test were never stable (see Figure 26). Constant adjustments of the flaps were required over the course of the test to keep the temperature within range. This would lead to time where the temperatures were briefly outside of the desired range.

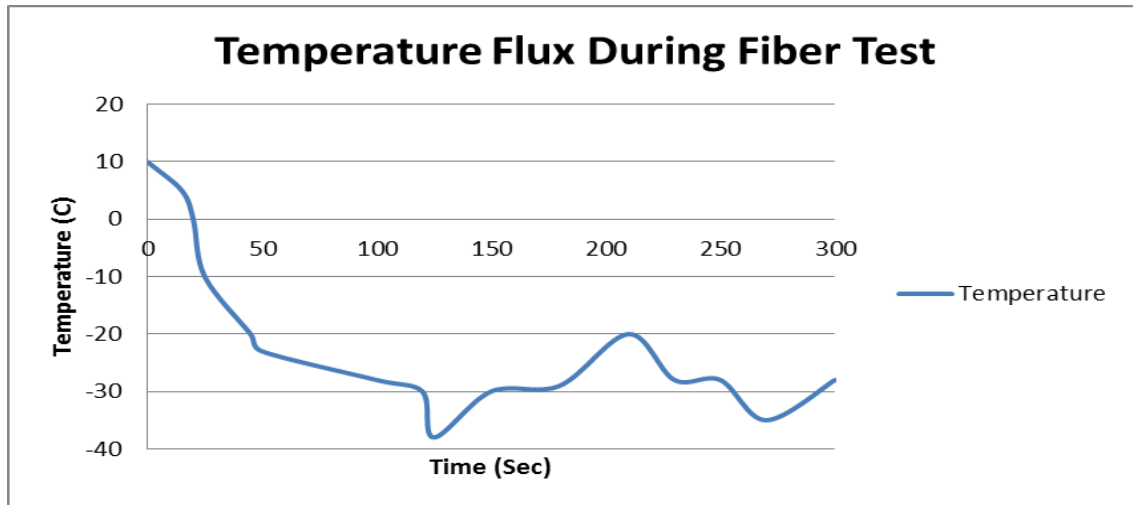


Figure 26. Sample temperature change over course of fiber test.

E. DISCUSSION

1. Data Distortion

a. *Temperature Effects*

In both the resin and composite tests, there was one test that demonstrated results closer to those of room temperatures than cold. This was likely caused by the temperature gradient inside the cold chamber and placement of the thermal couple. It is likely that the thermal couple ended up in a colder part of the chamber than the specimen. As a result of this, the specimen was tested at warmer temperatures than required. This resulted in the specimen producing data similar to that of room temperature. It is therefore acceptable to disregard those data points.

b. *Manufacturing Variance*

It was not possible to produce all of the resin specimens from the same batch of resin. As a result there was variation from batch to batch, and even specimen to specimen. Batch 2 was not tested because the specimen had a sticky surface upon curing. Resin Batch 3 performed significantly below that of other batches tested at room temperature. While all batches were mixed within the manufacturer's recommendations, the effects of excess release agent are unknown. It is possible that two of the batch 3 specimens had too much release agent along the sides of the mold, and it may have

resulted in early failure of two of the three samples. For this reason, the two early failures were excluded.

2. Anomalies and Theories

The fiber and resin preformed as expected for most materials exposed to extreme cold temperature. They suffered from a reduced ultimate strength and increased Young's modulus. The composite, however, had reduced yield strength and Young's modulus. This does not make sense, and was likely caused by a defect in the procedure.

a. Theory 1: Quiet Epoxy Failure

Other research has indicated that composites produce cracking and popping noises during the test that induce failure. [5], [9] These noises are caused by individual fibers separating from the resin and failing. If the epoxy failed as soon as the test started and did not produce noise, it would have distorted the strain earlier than thought. This would have resulted in an increase in the original length of the composite, and increase the calculated strain. This would severely lower the Young's modulus. The stress versus strain graph of Specimen 2-2 had an early abnormality compared to the graphs of other specimens (see Figure 27). This could have possibly been an early epoxy failure. While this failure was excluded from the Young's modulus calculation, it could have reduced the stiffness after this point. The Young's modulus was calculated after this point, because the failure occurred early in the test.

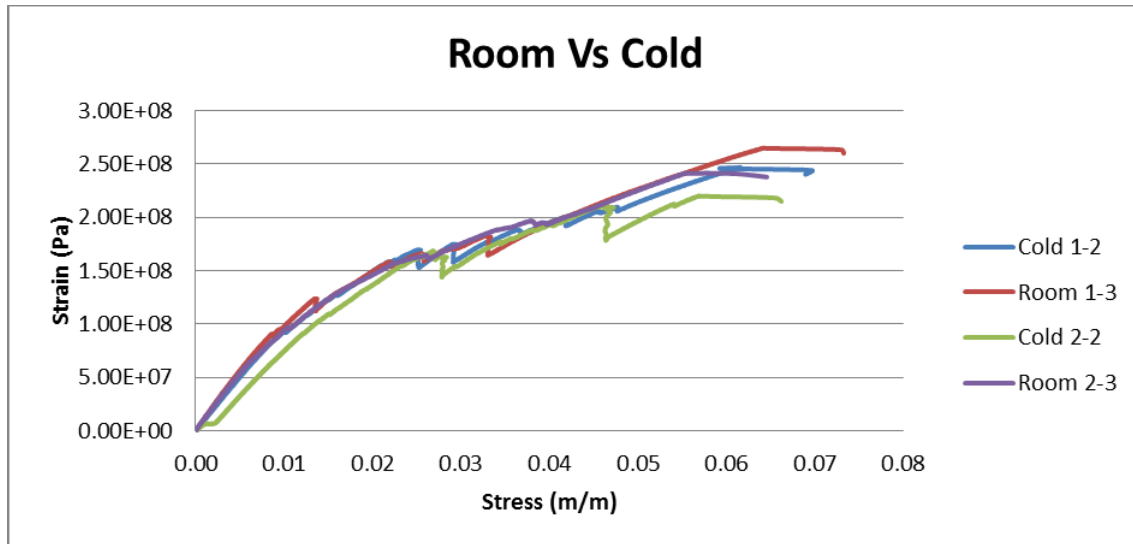


Figure 27. Comparison of room and cold composite stress versus strain graphs.

b. Theory 2: MTS Grip

Another potential source of error was the length of specimen contained within the MTS grip. Cutting the first set of tabs off the composite specimen resulted in the final specimen being 3 inches shorter than planned. As result, the cold specimen could not be completely gripped by the MTS. There was about 8 mm less specimen constrained by the grips during the cold tests. If Young's modulus was minimally affected by the cold, this could be enough to distort the results.

c. Theory 3: Accidental Damage

As discussed in the beginning, studies have shown composites suffer damage at low strain levels at cold temperatures and their Young's modulus is extremely affected by cyclic loading at arctic temperatures. Before conducting the test after cooling, the samples, the bottom MTS grip was released and reapplied to ensure there were no thermal contraction induced stresses. It is possible that this could have reduced Young's modulus due to cycling.

d. Theory 4: The Data is Correct

If the resin in the composite failed extremely early in the test, the strength of the composite would have been completely carried by the fiber. It is possible that with the

increase of the rigidity of the fiber, the load was not being carried evenly across the specimen and the overall Young's modulus would be decreased. With all the potential sources of error listed above, this cannot be proven with this data set. A new experimental setup would be required that eliminated the potential sources of error.

IV. CONCLUSION

The e-glass fiber used in this test was significantly affected by the colder temperature. While the 12% reduction in ultimate strength seems like it could be easily accounted for in design, the 50% increase in Young's modulus will cause issues. The increased stiffness decreases the ability of the fibers to work together, and this will result in non-constant performance by the e-glass fabric. The resin's reduction in ultimate strength of approximately 15% would make it appear to be a good match with the fiber in a composite, but 25% increase in Young's modulus could be a potential source of issues if the composite subjected to cold temperatures. The irregularities within the composite data set make it difficult to draw any firm conclusions, and if time and material had permitted, the experiment would have been rerun.

A. REPEATABILITY

The fiber and composite results did not demonstrate repeatability within their results. A proper fiber MTS holder and a different composite setup should help create repeatability. The resin samples did demonstrate repeatable if manufacturing inconsistencies and cold chamber variations were accounted for.

B. RECOMMENDATIONS

The cold chamber and fiber holder designs used in this experiment are not recommend for use in other experiments. The method used in the composite specimen could potentially work if a better adhesive was located, but there are additional recommend setups for tensile testing of composites [10]. It is recommended that dog bone shaped composite specimen be used in the future to eliminate the need for epoxy. It is also recommended that a larger sample size be prepared to account for manufacturing variations and testing difficulties.

C. FUTURE RESEARCH

While the results of the composite test were inconclusive and the fiber cold test had a source of error, the experiments still provide lessons that could be useful in future

research. By using the earlier recommendations, these tests could easily be completed with repeatable results. These tests could be included in research involving cyclic loading at Arctic temperatures. Other studies have shown that composites are highly affected by cyclic load, and this would be beneficial to the USN's composite patching research.

APPENDIX A. RESIN SPECIMEN FABRICATION

1. Cut piece of cardboard and wax paper to the size of mold.
2. Glue the cardboard and wax paper together to form a gasket.
3. Using top plate of mold as template, punch the hole pattern into the gasket.
4. Place the gasket in between the middle and bottom plates with the wax paper facing up.
5. Insert screws to fasten top and bottom plate together
6. Thread nuts onto 4 corner screws approximately 6.35 mm from the top.
7. Cut piece of bleeder cloth 2.5 times longer and 1.5 times wider than the mold.
8. On a glass table top, lay out a 610 mm by 610 mm box made of double sided sealing tape.
9. Place the end of the vacuum hose within the box and apply sealing tape as needed around hose where it enters into the box to create an air tight seal.
10. Cut out a square piece of Fibre Glast 1678 Strechlon 200 Bagging film slightly bigger than the tape box.
11. Apply a thin layer of petroleum jelly over all mold surfaces that will potentially be exposed to resin.
12. Mix 5 oz. of PRO-SET M1002 resin and 237 hardener at a weight ratio of 100:24 in a plastic mixing cup.

13. Place resin mixing cup in ultrasonic bath under a fume hood for five minutes to extract air bubbles from mix.
14. Using a 500 ml syringe inject resin into mold, and use the tip of the syringe to remove air bubbles from resin.
15. Place top plate onto four corner nuts.
16. Fold bleeder cloth in half and place it on top of plate, and ensure all four corners of the plate are covered with cloth.
17. Place vacuum plastic sheet over the top of table and ensure proper seal with sealing tape.
18. Turn on vacuum pump and reduce pressure to less than -33.8 kPa and leave resin mold at this pressure for 8 hours.
19. Remove vacuum sheet and let resin cure over night
20. Disassemble mold and extract specimens.

APPENDIX B. COMPOSITE SPECIMEN

1. Cut ten pieces 330 mm by 203 mm pieces of 7500 Hexcel 6 ounce plain weave e-glass fabric
2. Cut a 381 mm by 254 mm piece of bleeder cloth
3. Cut piece of ACP Composites V-20A Release Film Perforated slightly bigger than the bleeder cloth.
4. On a glass table top lay out a 610 mm by 610 mm box with double sided sealing tape.
5. Place the end of the vacuum hose within the box and apply sealing tape as needed around hose where it enters into the box to create an air tight seal.
6. Cut out a square piece of Fibre Glast 1678 Strechlon 200 bagging film slightly bigger than the tape box.
7. Tape a 457 mm by 330 mm piece of wax paper to the glass inside the tape box
8. Mix 5 oz. of PRO-SET M1002 Resin and 237 Hardener at a weight ratio of 100:24 in a plastic mixing cup.
9. Spread a layer of resin on the wax paper approximately the same size as the e-glass fabric rectangles.
10. Place first piece of E-glass on resin and use a roller to submerge the e-glass into the resin.
11. Apply remaining layers of E-Glass and add resin as necessary.
12. Place perforated film on top of wet e-glass.

13. Place bleeder cloth over perforated film.
14. Place bagging film over the top of table and ensure proper seal with sealing tape.
15. Turn on vacuum pump and reduce pressure to less than -33.8 kPa and leave composite at this pressure for 8 hours.
16. Remove vacuum sheet and let composite cure over night
17. Peel off perforated film
18. Cut composite to desired size

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